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STATIC FATIGUE OF A SILICONIZED SILICON CARBIDE

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ABSTRACT

The static fatigue resistance of a novel siliconized silicon carbide has been evaluated. The material is fabricated by the in-situ reaction of silicon with fine, uniform carbon skeletons produced from liquid polymer solutions. The process has potential to make net shape parts without final machining. A very fine microstructure results which, in turn, gives high strength. Flexural specimens tested in this study had high strength at room and elevated temperatures (up to 1300 C), and had good static fatigue and creep resistance.

(Keywords:)

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INTRODUCTION

This work was part of a Department of Energy sponsored program to perform screening tests for room temperature strength, static fatigue, and creep resistance of new heat engine structural ceramics. Previous reports have been prepared for a wide variety of silicon carbide, silicon nitride, and zirconia materials.[1-10] In particular, References 6 and 10 include static fatigue and creep results for the siliconized silicon carbide family of materials.

The siliconized silicon carbide described in this report was prepared at the University of Michigan under contract (DAAG46-80-C-0056) to the Defense Advanced Research Agency. Its development is documented in a series of technical reports.[11-14] These reports had a limited distribution and therefore, a detailed introduction on materials processing is included herein. This material was developed using novel techniques intended to produce a uniform and very fine microstructure. The size of strength-limiting flaws is often commensurate with microstructural features. It was hoped that a finer and more uniform microstructure would lead to higher strength than heretofore reported for siliconized silicon carbide. The fabrication process is a novel variation of reaction bonding of silicon with carbon.[11-14] The key to this process is obtaining a uniform pure carbon skeleton which is made by casting a low viscosity liquid polymer that is subsequently carbonized. This carbon body is then infiltrated and reacted with silicon metal to form a silicon-silicon carbide composite. The carbon body structure can be accurately controlled by variation of polymer concentration, and polymerization times and temperatures. Conventional powder processes such as grinding, mixing, and pressing are not involved.[11-14]

Preliminary work established that refining the structure of the carbon skeleton results in a finer final microstructure and that bulk density, pore, and carbon size distributions were key variables.[11,12] A skeleton density of 0.87 g/cm^3 was optimum. Higher values lead to incomplete silicon infiltration, whereas lower values cause strength reduction. Pore size uniformity is crucial so that silicon infiltration is uniform. An optimum pore size of 2 microns permitted up to 15 cm x 10 cm x 5 cm to be infiltrated in 1-2 hours. Carbon size is controlled to prevent skeleton sections that are too thick to react completely or which form coarse silicon carbide zones. Carbon "solid size" should be less than 10 microns. Green bodies with pore and carbon size less than 1 micron have also been produced. Silicon infiltration temperatures were between 1450°C and 1575°C. Residual-free silicon content ranged from 5% to 20%, with 10% typically the goal.[13]

The polymer precursor can be cast or machined prior to carbonization. Carbonization will cause shrinkage, but of a reproducible magnitude. However, warpage during carbonization has to be controlled. In principle, complex shapes can be fabricated by this process, although most of the effort was on plates or rectangular bars.

Processing problems include microstructure coarsening, silicon lake formation, nonuniform surface reaction layers, and incomplete carbon reaction. These difficulties must be overcome by careful control of processing parameters.

Previous mechanical property evaluations have been confined to room temperature flexure strength and fracture toughness.[11-14] Four-point flexure strength averages were as high as 714 MPa (105 ksi) with Weibull moduli in the 4-8 range.[13] Fracture toughness was estimated from a Vickers indentation method to have been in the 3.5 $MN/m^{1.5}$ range.[13]

MATERIAL CHARACTERIZATION

Forty rectangular bars, nominally 2.9 x 3.1 x 50.8 mm in size, were prepared for this study.* These bars were in an as-processed form and had surface irregularities so that they had to be machined to a final size of 2.1 x 2.5 x 50.8 mm. The average bulk density of the machined bars was 3.10 g/cm^3 with a 0.02 standard deviation.

Metallographic inspection of several polished sections revealed a range of structures as shown in Figures 1a through 1c. The free silicon content is approximately 20 percent. A quantitative estimate by point or lineal analysis could not be made since the silicon (white phase) actually contains a very fine dispersed silicon carbide structure which would be hard to resolve. For comparison, Figure 2 is a polished section of NC 435 siliconized carbide, which is the finest grain material in the NC 430, NC 433, and NC 435 family.* Residual carbon in the University of Michigan material can be eliminated by high temperature heat treating, although such treatment can lead to microstructure coarsening. The residual carbon is reasonably uniform in size (~10 μm) and is spherical. A chemical analysis by inductive coupled argon plasma spectroscopy is shown in Table 1. Elemental impurity levels are typical for siliconized silicon carbide.[5]

Table 1. ELEMENTAL IMPURITY ANALYSIS

					Weight Percent						
AL	B	Ca	Co	Cr	Cu	Fe	Mg	Mn	Ni	Ti	Zr
0.09	0.27	0.06	*	0.009	0.13	0.11	0.015	0.031	*	0.025	0.002

*Not Detected

EXPERIMENTAL PROCEDURE

Room temperature strength was evaluated to serve as a reference strength and to determine the nature of strength-limiting flaws. Four-point flexural testing was performed with B configuration (20-mm x 40-mm inner and outer spans) prescribed by MIL-STD-1942 (MR), but the final specimen size (2.1 x 2.5 x 50.8 mm) was not in accordance with the standard due to processing constraints.

High temperature flexural stress rupture and stepped temperature stress rupture (STSR) testing were performed to assess the static fatigue and creep resistances. Isothermal stress rupture experiments were performed at 1200°C in air for comparison to previous results.[1-10] STSR experiments[15] were under deadweight loading, but with each specimen exposed to a range of temperatures from 1000°C to 1300°C. A similar schedule has been used for the testing of other siliconized carbides: 24-hour holds at 1000°C, 1100°C, and 1200°C, and a 72-hour hold at 1300°C. The purpose of this procedure is to quickly assess the temperature and stress levels that static fatigue and creep processes are active. The applied stresses were computed on the basis of the elastic formulation.

*University of Michigan Lot 332-22.
†Norton Company, Worcester, MA, see Reference 5 for additional details.

Figure 1a. Polished section illustrating the silicon carbide (grey), silicon (white), and carbon/void (black) phases.

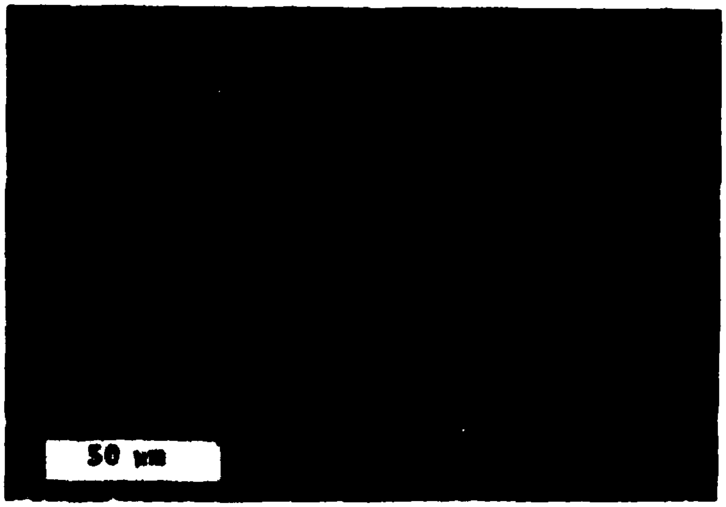


Figure 1b. Higher magnification showing that the white phase is actually complex silicon/silicon carbide structure.

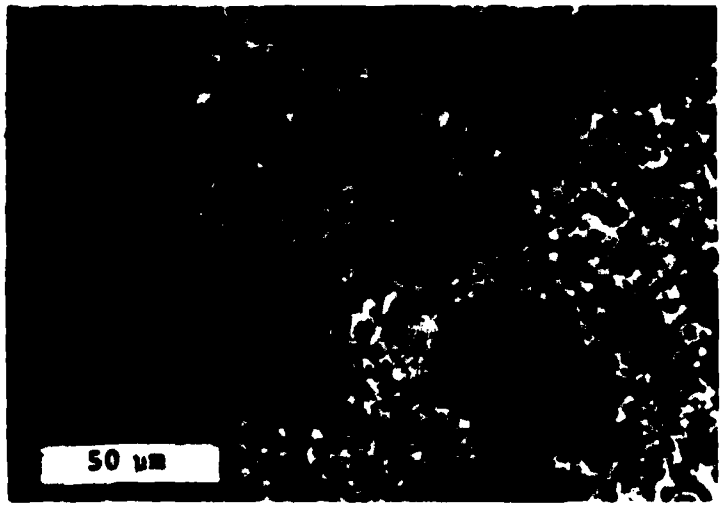
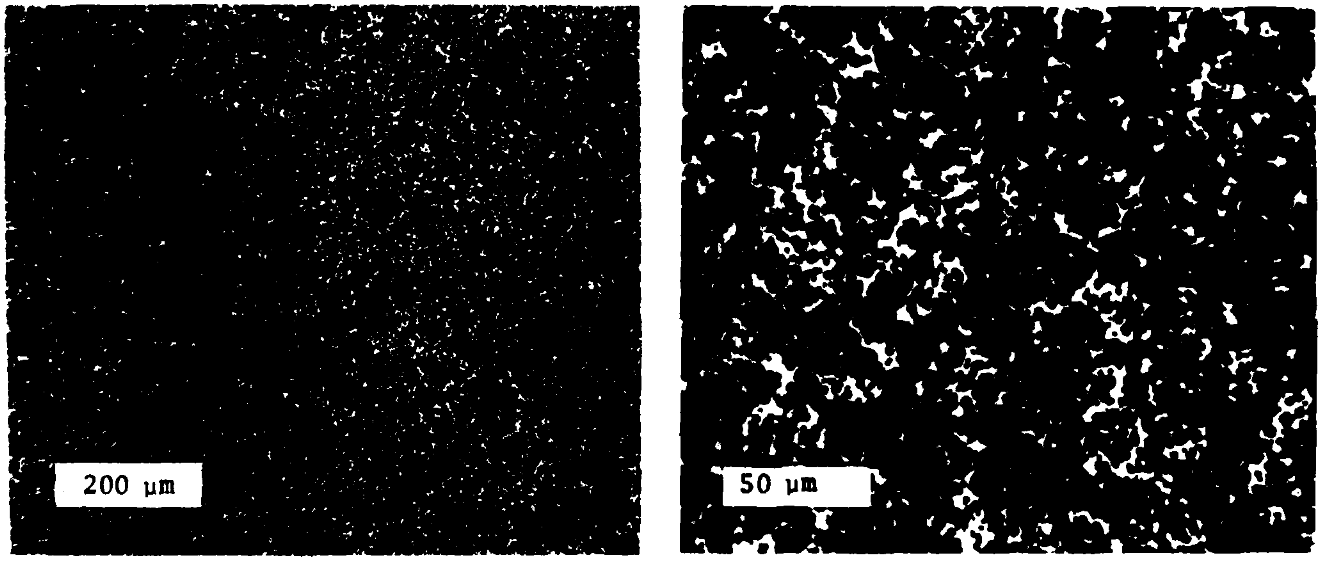


Figure 1c. A zone with excessive silicon carbide grain growth.

Three new high temperature, hot-pressed silicon carbide fixtures were used for this study (Figure 3). The fixture is designed for well-machined specimens and has inner and outer spans of 20 mm and 40 mm, respectively. The inner span loading is through fixed knife edges; the outer span, through rollers that were permitted to roll during a test in order to minimize friction constraints. The rollers were butted against a shoulder at room temperature and tacked in place with a household cement (which subsequently burns off). The specimen and upper load block are inserted and a slight preload applied prior to heating in the furnace. This design is an interim one, pending a more sophisticated jig that has inner span rollers as well.

Specimens which survived stress rupture or STSR testing intact were subsequently measured for weight change, final creep strain, and retained strength. The creep strain was determined by photographic enlarging the inner span curvature, and measuring the midspan deflection relative to the inner span points.* Retained strength was measured at room temperature with the MIL-STD-1942 (MR) B fixtures.



(a) (b)

Figure 2. Polished sections of NC 435. Silicon carbide is the grey phase and silicon is in the white phase.

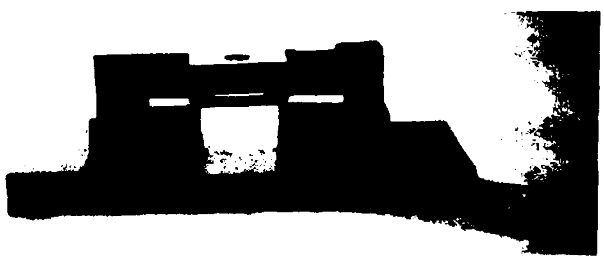


Figure 3. High temperature test fixture. A white alumina specimen is in place.

*Strain = (4 x specimen thickness x midspan deflection)/(inner span length)^2.

RESULTS

The average room temperature strength for 16 specimens was 609 MPa (88 ksi). One very low strength specimen (174 MPa) fractured from a large crack already present (but not noticed prior to testing) as shown in Figure 4. If this datum is deleted, the average strength was 638 MPa (92.5 ksi) with a 144 MPa (21 ksi) standard deviation. The highest strength was 805 MPa (117 ksi). A Weibull two-parameter analysis is shown in Figure 5. The strength values were ordered and assigned probabilities of i/n+1 where i is the ith datum and n is the total number of specimens. A least-square fitted line matched the data very well with a slope of 7.5 and a characteristic strength of the bend specimen of 678 MPa.

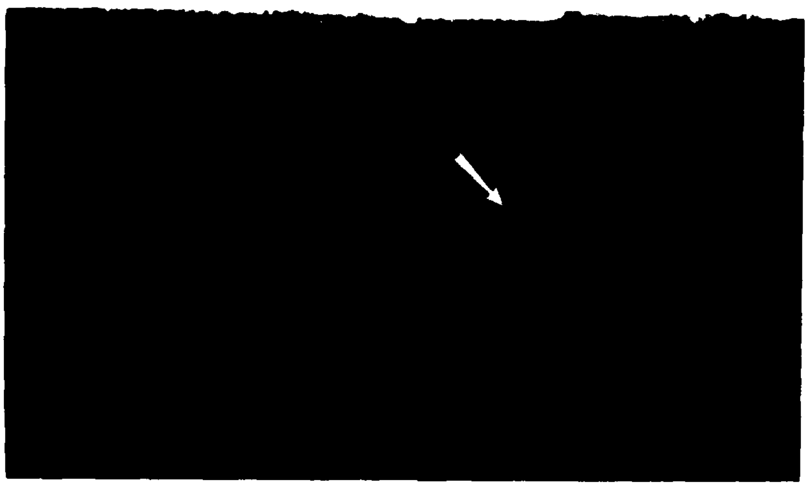


Figure 4. Tensile face of a flexure specimen which fractured at very low strength (174 MPa). The arrow highlights the origin of failure, which is the crack, even though it is oriented 90° to the applied stress.

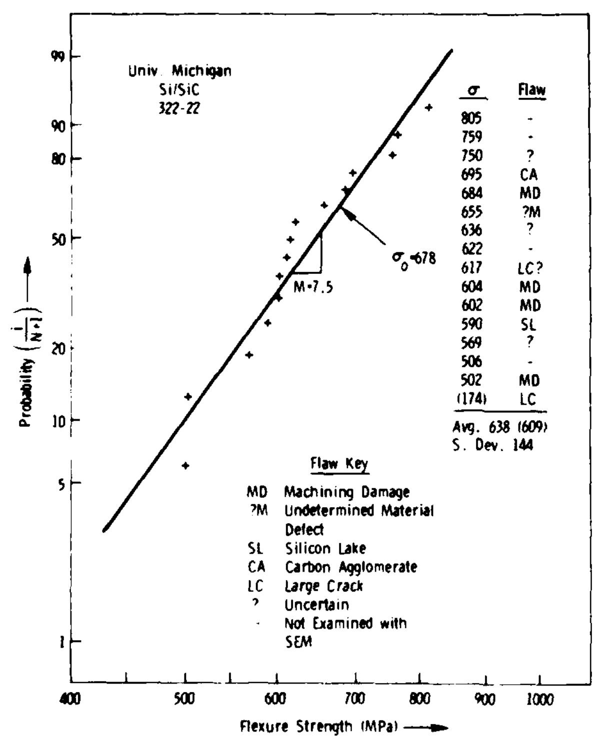


Figure 5. Weibull two-parameter representation of the room temperature reference strengths.

The fracture surfaces had small mirrors which facilitated location of the strength-limiting flaws which unfortunately were too small to characterize optically. A representitive group was examined with the SEM which revealed a range of possible flaw types including: machining damage, carbon agglomerates, silicon agglomerates, and sometimes, planar seams of material different in microstructure than the bulk (see Figures 6 and 7). In two instances, it was possible to estimate the fracture toughness K_{Ic} based upon the machining flaw size and shape and failure stress (assuming no residual stress). Values of 3.1 and 3.2 $MN/m^{1.5}$ were obtained. The best estimates of the flaw type are listed on the side of the Weibull plot (Figure 5). Similar defects have been reported previously in the progress reports on the development of this material.[13,14]

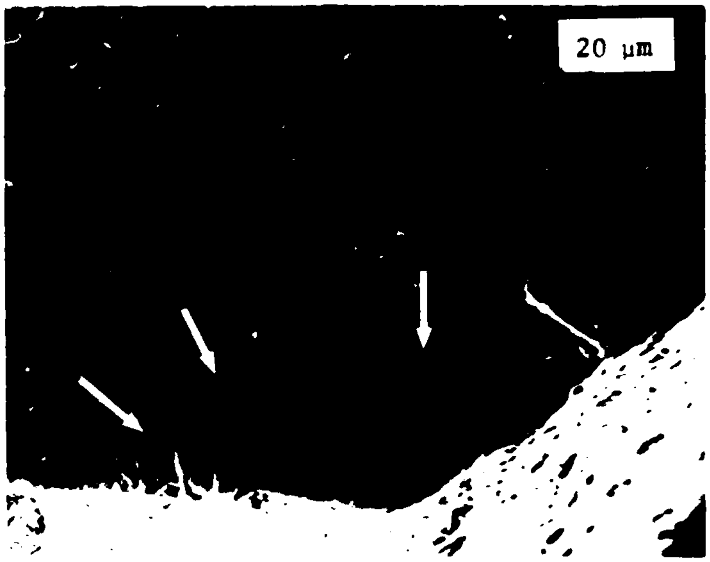


Figure 6. SEM photo of the fracture surface of a specimen revealing the strength-limiting defect is machining damage.

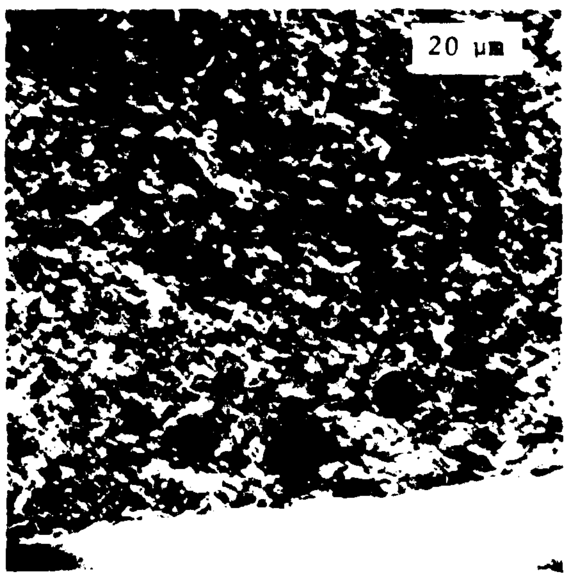


Figure 7. SEM photo of the fracture surface revealing three carbon agglomerates which were strength limiting.

STSR results are given in Figure 8. Three specimens failed in a time-dependent manner, two failed immediately upon loading at 1000°C, and three specimens survived intact. The results suggest that time-dependent failure can occur, but only for highly stressed specimens in the 1000°C to 1300°C range. The fracture origin for the specimen which broke at 0.04 hour at 1000°C was a material defect, probably a pore or a region originally carbon-rich. The origin looked like a fast fracture site and had no slow crack growth (SCG) markings. The 530 MPa specimen, which failed at one hour at 1200°C (after accumulating 24 hours at 1000°C and 1100°C) had machining damage at the origin and also did not have clear time-dependent markings (Figure 9). The fracture surface on the third time-dependent failure, 0.15 hour on the 1300°C step on a 500 MPa stressed specimen, was heavily damaged by impact on the flexural fixtures.

The three STSR survivors had negligible changes in mass (0.26% loss to 0.03% gain). Permanent creep strains were small; 0.20%, 0.13%, and 0.19% for the specimens loaded to 350, 400, and 500 MPa, respectively. Retained strengths at room temperature were 639, 617, and 689 MPa.

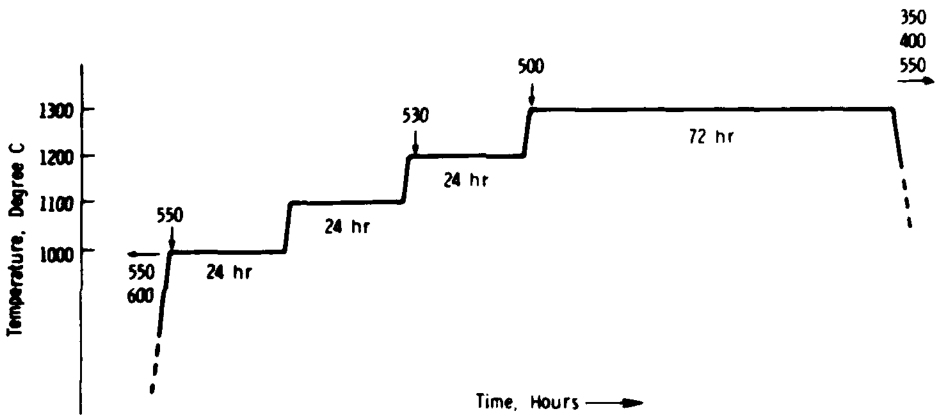


Figure 8. STSR results. Each arrow denotes one (or more) specimen and is labeled with the applied elastic stress in MPa.

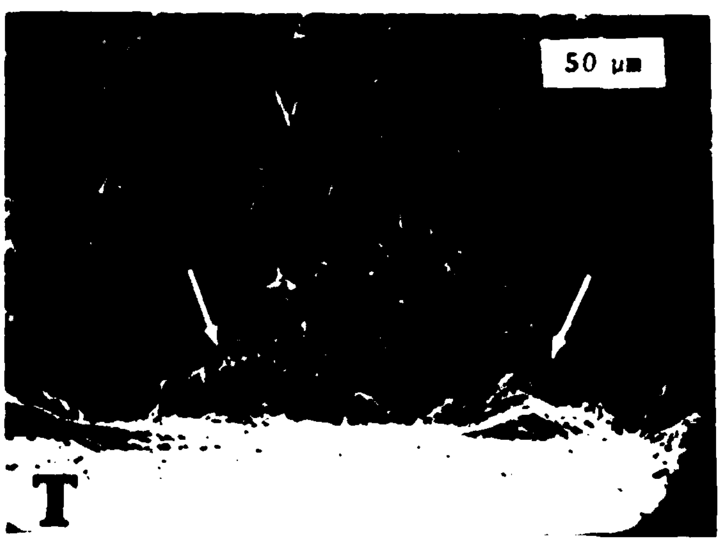


Figure 9. SEM photo of the fracture origin of an STSR specimen which failed at one hour in the 1200°C step. The arrows highlight machining damage.

Isothermal stress rupture experiments at 1200°C are given in Figure 10. Two specimens failed upon loading (arrows left), four failed in time-dependent manner (solid points), and five did not fail (arrows right). One of the latter accumulated 8559 hours (0.98 year) whereupon a furnace malfunction occurred causing temperature to soar to 1725°C. The specimen failed and there was excessive melting and oxidation of the free silicon. The fracture surfaces of the four time-dependent failures were difficult to interpret, sometimes due to excessive impact damage sustained when the specimen struck the fixture after fracture. In one instance, the origin was a zone or seam of material different than the bulk (Figure 11). The origin of the specimen which failed at 2400 hours was a surface-connected pore associated with a zone of microporosity. The three 550 MPa, 1000-hour survivors and the 600 MPa survivor had negligible mass changes from 0.2% loss to 0.04% gain. Permanent creep strains were 0.32%, 0.33%, 0.29% and 0.21%, respectively. The room temperature retained strengths were generally higher than the reference strength (638 MPa); 679, 624, 709, and 966 MPa, respectively.

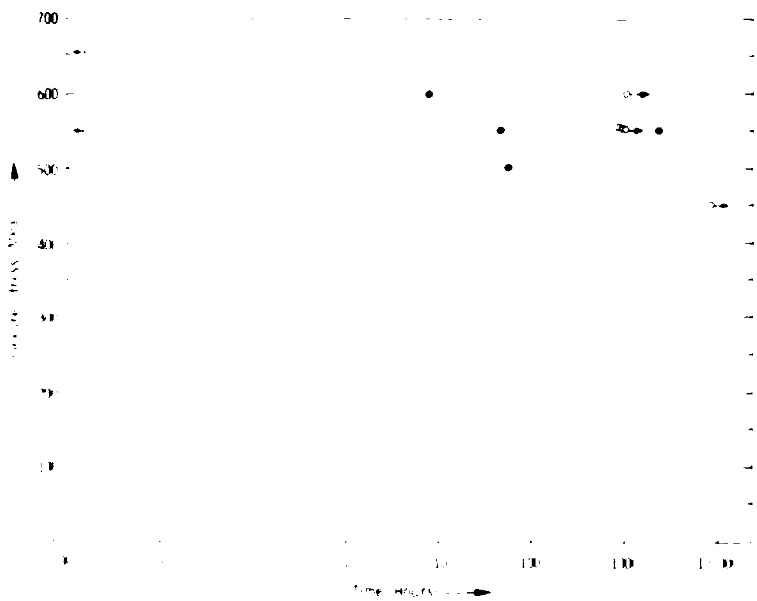


Figure 10. Flexural stress rupture at 1200°C.

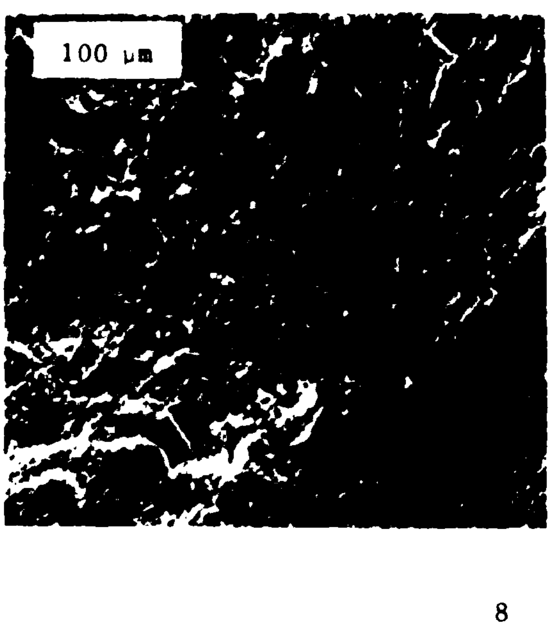


Figure 11. Fracture origin of specimen which failed at 58.5 hours at 1200°C with 500 MPa loading.

Both the STSR and 1200°C stress rupture survivors had a coherent thin surface oxide layer that was interrupted by numerous small spherical pores. These presumably result from the oxidation of surface-connected, spherical carbon pockets such as shown in Figure 1.

DISCUSSION

This material is the latest in a series of siliconized silicon carbides dating back to the REFEL grades[14] and more recently, the NC 430 series[15] and the Silcomp grades.[16] The University of Michigan material uses a novel process involving carbonization of a polymer precursor in order to achieve a much finer microstructure. In principle, this should result in higher strength if strength-limiting flaws scale in size with microstructural features. The results in this report substantiate this approach. The University of Michigan material has the highest strength reported for siliconized silicon carbide and also the finest scaled microstructure. Room temperature flexural strengths reported herein are in good agreement with those presented in the earlier progress reports on this material's development.[11-14] Unfortunately, to maintain consistently high strength, the uniformity of the microstructure must be maintained. This is a common and difficult problem in the siliconized silicon carbide family.[5,14] More careful processing can presumably alleviate this problem, but the presence of coarser surface reaction layers may be difficult to overcome. The low Weibull modulus (7.5) is also consistent with earlier findings[13,14] and is partly a consequence of a nonuniform microstructure. Very complex multiple flaw populations no doubt contribute to the strength scatter as well.

The flexure specimen preparation in this study was fairly careful and induced subsurface cracks of rather small size (~10 microns). Nevertheless, such flaws often were strength limiting, because of low inherent fracture toughness (~3 $MN/m^{1.5}$) of siliconized silicon carbide. The implication is that if the microstructure was made even finer and more uniform, machining preparation would limit strength. If specimens were not machined and were tested in an as-processed shape, then surface irregularities (common to siliconized silicon carbide) would limit strength. Thus, the practical upper limit of strength for siliconized silicon carbide may indeed be in the 600-800 MPa range. In any case, the University of Michigan grade evaluated in this report has achieved this practical limit. Any further improvements should be directed at improving the Weibull modulus, eliminating surface coarsened regions, or improving fracture toughness.

The University of Michigan material has high static fatique and creep resistance. This is common to siliconized silicon carbides wherein time-dependent failures in the 1000°C to 1300°C range only occur at stresses close to the fast fracture strength. Thus, since the University of Michigan material has a very high reference strength, it is able to sustain very high flexural stresses up to 1300°C. In this context, the material outperforms many other silicon carbides and nitrides.[5,6] The few time-dependent fractures had unclear fracture origin features making identification of a mechanism of static fatique difficult. Creep fracture could be ruled out due to the small strains (0.2%) involved, unlike NC 435, the finest grain member of the NC 430 series.[5,6] Indeed, a comparison of the coarser NC 433 to NC 435 showed the latter was more sensitive to slow crack growth and creep fracture.[5] The higher creep rate in the finer grained material was not surprising. It may therefore be expected that the University of Michigan material which possesses an even finer structure, would have higher creep strains. It did not, despite the very high applied stresses. The creep resistance of this material, thus, is superior to the other siliconized silicon carbides and many other silicon carbides and nitrides in the 1000°C to 1300°C range.

The specimens which survived the stress rupture and STSR exposures had no strength degradation. Indeed, one such specimen had the highest strength measured in this study (966 MPa).

CONCLUSION

A new siliconized silicon carbide, fabricated by a novel processing technique, has demonstrated very high flexural strength. This is a consequence of the processing method which produces a very fine microstructure. This material probably had recorded the maximum practical fast fracture strength for the siliconized silicon carbide family of materials. Strength scatter is somewhat high, which is typical of siliconized silicon carbides due to microstructural nonuniformity and multiple flaw populations. Static fatique and creep resistances were very high and are superior to other siliconized silicon carbides, and many silicon carbides and nitrides in the 1000°C to 1300°C range. Failures only occurred for specimens loaded nearly to the fast fracture strength.

ACKNOWLEDGMENT

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The static fatigue resistance of a novel siliconized silicon carbide has been evaluated. The material is fabricated by the in-situ reaction of silicon with fine, uniform carbon skeletons produced from liquid polymer solutions. The process has potential to make net shape parts without final machining. A very fine microstructure results which, in turn, gives high stength. Flexural specimens tested in this study had high strength at room and elevated temperatures (up to 1300°C), and had good static fatigue and creep resistance.